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IS 5298 (1983): Method for determination of distillation range and of distillation yield [CHD 1: Inorganic Chemicals]



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IS : 5298 - 1983

( Reaffirmed 2010 )

*Indian Standard*

**METHOD FOR DETERMINATION OF  
DISTILLATION RANGE AND  
DISTILLATION YIELD**

**( *First Revision* )**

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**INDIAN STANDARDS INSTITUTION  
MANAM BHAVAN, 9 BAHADUR SHAH ZAFAR MARG  
NEW DELHI 110002**

# *Indian Standard*

## METHOD FOR DETERMINATION OF DISTILLATION RANGE AND DISTILLATION YIELD

### ( *First Revision* )

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( *Continued on page 2* )

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***Indian Standard***  
**METHOD FOR DETERMINATION OF  
DISTILLATION RANGE AND  
DISTILLATION YIELD**  
**( *First Revision* )**

**0. FOREWORD**

0.1 This Indian Standard was adopted by the Indian Standards Institution on 15 June 1983, after the draft finalized by the Chemical Standards Sectional Committee had been approved by the Chemical Division Council.

0.2 Distillation range is determined to identify an unknown substance, to measure approximate purity, or to ascertain whether or not a substance conforms to a specification. In all these cases, reproducibility of results of distillation range depends on close adherence to details, both as regards the apparatus and the procedure. Therefore, this standard was first issued in 1969, covering an apparatus which was suitable for the determination of distillation range of most of the liquid chemicals — both organic and inorganic.

0.2.1 This standard has now been revised, on the recommendations of the Ministry of Defence, to include provisions for ground glass interchangeable joints, temperature ranges of the water to be circulated, and cooling of the sample and the distillate. Amendment No. 1 issued in January 1975 has also been incorporated in the present revision.

0.2.2 For materials, for which this apparatus is not suitable, the details of the appropriate apparatus and procedure would be covered in the individual material specifications.

0.3 Reference should be made to the relevant specification ( IS : 2480-1973\* ) regarding details of the thermometer to be used, for example, whether calibrated for total immersion, dimensions of bulb, etc. Further, greatest precaution should be taken to ensure that liquids that may form peroxides ( like others ) are not distilled.

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\*Specification for general purpose glass thermometers ( *first revision* ).

**0.4** While revising this standard, assistance has also been derived from the following:

ISO 383-1976	Laboratory glassware — Interchangeable conical ground joints. International Organization for Standardization.
BS 572 : 1980	Interchangeable conical ground joints. British Standards Institution.
ASTM D 1078	Distillation range of volatile organic liquids. American Society for Testing and Materials.

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## 1. SCOPE

**1.1** This standard prescribes methods for the determination of distillation range and distillation yield of liquids in general.

NOTE — This standard is applicable, as a general method, for liquids other than petroleum distillates for which IS : 1448 [P:18]-1967\* shall be applicable.

## 2. TERMINOLOGY

**2.0** For the purpose of this Standard, the following definitions shall apply.

**2.1 Distillation** — It is the process of vaporizing a liquid and collecting its vapour, which is usually condensed to a liquid. Pure substances may be distilled, but the process is usually used to achieve separation of liquid mixtures because of the difference in composition between the liquid and the vapour formed from it.

**2.2 Distillation Range** — The distillation range of a substance is the range of temperature within which a specified portion distills.

**2.3 Distillation Yield** — The volume of distillate collected in the receiver within the corrected specified distillation temperatures ( distillation range at 760 mm Hg pressure and expressed as the percentage by volume ).

**2.4 Initial Boiling Point** — That temperature reading which is observed at the instant the first drop of condensate falls from the lower end of the condenser tube.

**2.5 Final Boiling Point** — Maximum temperature reading obtained during the test; this usually occurs after the evaporation of all liquid from the bottom of the flask. The term 'maximum temperature' is a frequently used synonym.

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\*Methods of test for petroleum and its products: [ P : 18 ] Distillation ( *first revision* ).



**2.6 Dry Point** — The thermometer reading which is observed at the instant the last drop of liquid evaporates from the lowest point on the flask. Any drops or films of liquid on the side of the flask or on the thermometer are disregarded.

### 3. PRINCIPLE OF THE METHOD

**3.1** The sample ( 100 ml ) is distilled under the prescribed conditions and either the volume of distillate collected over a specified range of temperature, or alternatively the initial boiling point and dry point are determined.

### 4. APPARATUS

**4.0 General** — All apparatus used should be thoroughly cleaned and dried. The apparatus shall be assembled as shown in Fig. 1. However interchangeable ground glass joints, where available, may also be used as per the details given below:

Name of Joint	Designation	Diameter of Ground Zone		Axial Length of Ground Zone
		Large End	Small End	
		(mm)	(mm)	(mm)
Flask to thermometer	B 14	14.5	12.2	23
Flask to condenser	B 19	18.8	16.2	26
Condenser to delivery tube	B 19	18.8	16.2	26

**4.1 Distillation Flask** — See IS : 2620-1963\*.

**4.2 Thermometer** — of total immersion type and of appropriate range conforming to IS : 2480-1973†.

**4.3 Receiver** — 100-ml capacity, complying with Fig. 2.

**4.4 Condenser** — of glass, shown in Fig. 3, the inner tube having the following dimensions:

Internal diameter	14.0 ± 1.0 mm
Wall thickness	1.0 to 1.5 mm

\*Specification for distilling flasks.

†Specification for solid stem general purpose glass thermometers ( first revision ).

Length of shorter limb	$55 \pm 5$ mm
Length of straight portion of longer limb	$600 \pm 10$ mm
Angle inclined between longer and shorter limb	$87 \pm 3^\circ$

**4.4.1** The inlet of the inner tube of the condenser is finished square with its axis, and its outlet smoothly ground at an angle of approximately  $45^\circ$  with the axis of the tube at that point, as shown in Fig. 3. The central straight portion of the water jacket ( see Fig. 3 ) is  $450 \pm 10$  mm in length, and its external diameter  $35 \pm 3$  mm.

**4.4.2 Air Condenser** — For liquids having boiling point above  $150^\circ\text{C}$ , an air condenser of suitable size shall be used.

**4.5 Draught Screen** — Rectangular in cross-section and open at the top and bottom. It has the dimensions shown in Fig. 4 and is made of sheet-metal 0.8 mm thick.

**4.5.1** In each of the two narrower sides of the draught screen there are two circular air vent holes 25 mm in diameter, situated below the asbestos shelf, as shown in Fig. 4.

**4.5.2** In each of the four sides of the draught screen are the three air vent holes with their centres 25 mm above the base of the draught screen. These holes occupy the positions shown in Fig. 4, the diameter of each of the holes centrally situated in the wider sides being 25 mm and the remaining ten holes 12.5 mm in diameter.

**4.5.3** At the middle of each of the wider sides, a vertical slot for the side tube of the distillation flask, and dimensions as shown in Fig. 4 is cut downward from the top of the screen. A removable shutter conforming to the dimensions in Fig. 5 is provided for closing whichever vertical slot is not in use.

**4.5.4** A shelf of hard asbestos board 6 mm in thickness and possessing a centrally cut circular hole 110 mm in diameter, is supported horizontally in the screen and fits closely to the sides of the screen to ensure that hot gases from the source of heat do not come in contact with the sides or neck of the flask. The supports for this asbestos shelf may conveniently consist of triangular pieces of metal sheet firmly fixed to the screen at its four corners.

**4.5.5** In one of the narrower sides of the screen, a door is provided having dimensions shown in Fig. 4 and overlapping an opening in the screen, by approximately 5 mm all round.

**4.5.6** In each of the narrower sides of the screen, a mica window is placed centrally with the bottom of the window level with the top of the asbestos shelf. The dimensions and positions of the windows are shown in Fig. 4.

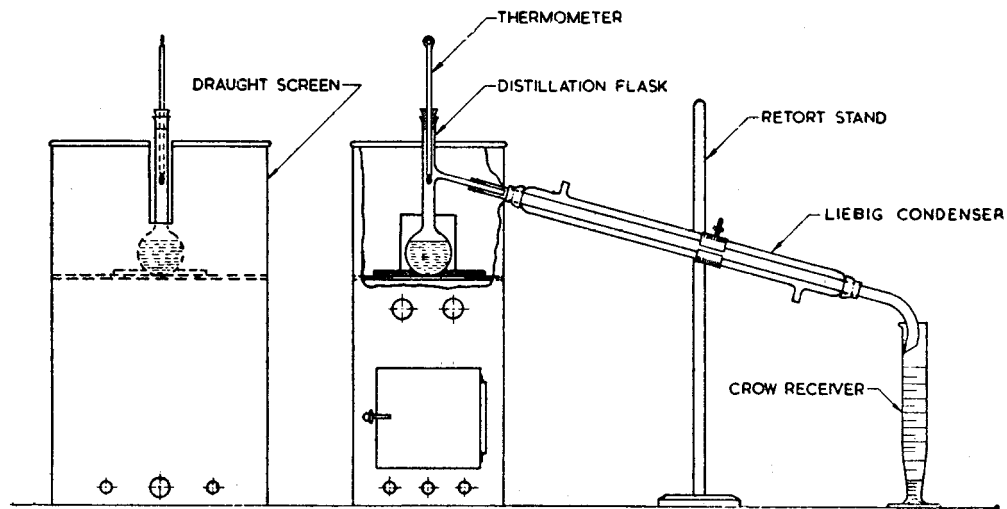
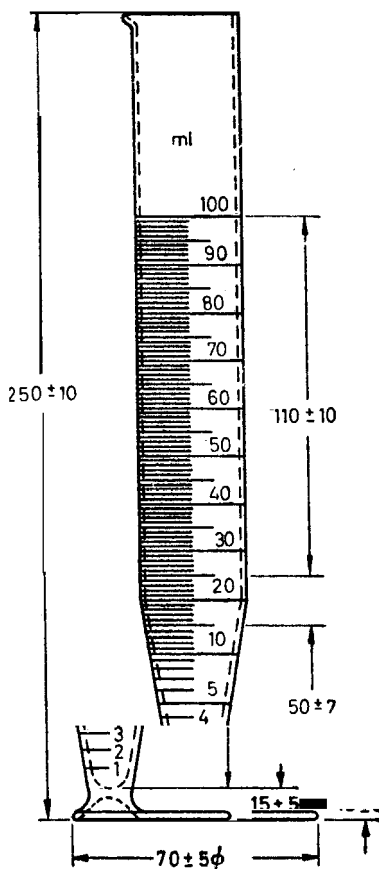


FIG. 1 ASSEMBLY OF DISTILLATION APPARATUS

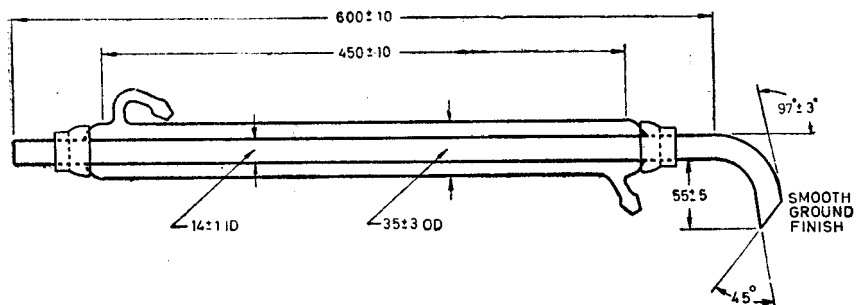


All dimensions in millimetres.

FIG. 2 DISTILLATION RECEIVER

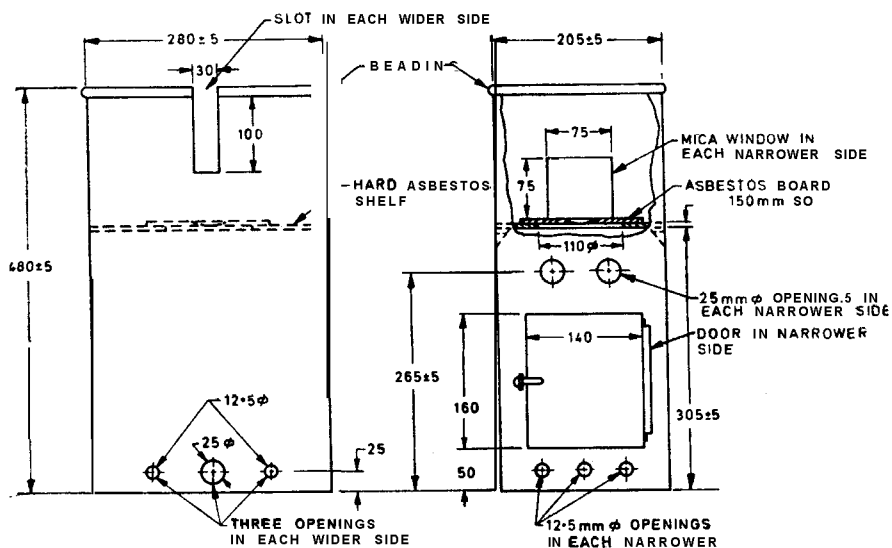
**4.6 Asbestos Board** — In addition to the asbestos shelf referred to in the previous section, an asbestos board 150 mm square and 6 mm thick is required. For the distillation of liquids boiling below 60°C the diameter for hole shall be 30 mm and for others it shall be 50 mm.

**4.7 Source of Heat** — An adjustable gas burner or electric heater so adjusted as to distil the product at the uniform rate specified in 7.3.



All dimensions in millimetres.

FIG. 3 CONDENSER

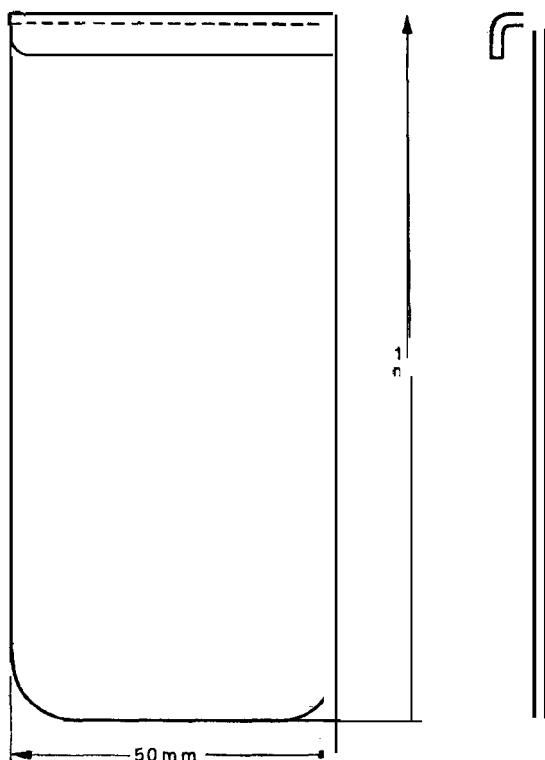


All dimensions in millimetres.

FIG. 4 DRAUGHT SCREEN

## 5. ASSEMBLY OF THE APPARATUS

5.0 The apparatus is assembled as shown in Fig. 1, and attention is called to the details given in 5.1 to 5.3.



All dimensions in millimetres.

FIG. 5 REMOVABLE SHUTTER FOR THE DRAUGHT SCREEN

5.1 Position of the Thermometer — The thermometer is held in the neck of the flask by means of a B 14 ground glass joint or a well-fitting stopper of a material which is not affected by the liquid to be distilled. The top of the bulb joint shall be at least 5 mm below the lower edge of the side-tube

5.2 Support for the Flask — The asbestos board ( see 4.6 ) is placed on the top of the asbestos shelf of the draught screen so that the two holes are approximately concentric. The flask is then placed in position and pressed down so as to close completely the hole in the asbestos board.

5.3 Connection of Flask to Condenser — The flask is connected to the condenser by means of a B 19 ground glass joint or tight-fitting cork

mounted on the side tube so that the end of the side tube projects at least 25 mm into the condenser and is coaxial with it.

## 6. ADJUSTMENTS TO BE APPLIED TO SPECIFIED TEMPERATURES BEFORE COMMENCING THE DISTILLATION ( FOR YIELD )

**6.1 Adjustments for Barometric Pressure** — When the barometric pressure ( corrected as in 11 ) deviates from 760 mm Hg, apply adjustments to the specified distillation temperatures, as indicated in the specification for the material under test. It should be noted that these adjustments are valid only for pressures above 700 mm Hg.

**6.2 Adjustments for Thermometer Error** — If the thermometer gives incorrect readings at the specified distillation temperature adjusted in accordance with 6.1, further adjust the temperatures by adding the amount of error if the thermometer is reading high or subtracting the amount of error if the thermometer is reading low.

## 7. PROCEDURE

**7.1** Measure 100 ml of the sample in the receiver at the temperature specified for different boiling ranges as indicated below:

<i>Initial Boiling Point ( °C )</i>	<i>Sample Temperature ( °C )</i>
Up to 70	Below 20
70 and above	Ambient temperature

Transfer the sample directly to the distillation flask, allowing the receiver to drain for 15 to 20 seconds. Add a few small pieces of clean, dry porous pot or pumice stone.

NOTE — For viscous liquids, a longer drainage period may be necessary, but the drainage time should not exceed 5 minutes. Do not allow any of the samples to enter the vapour tube.

**7.2** Assemble the apparatus as instructed in 5 and Fig. 1. Place the receiver, without drying, at the outlet of the condenser tube in such a position that the condenser tube extends into the receiver at least 25 mm but does not extend below the 100 ml-mark. Regulate the temperature of the condenser water and of the bath in which the distillate collection receiver is placed as indicated below:

<i>Initial Boiling Point of Distillate ( °C )</i>	<i>Water Temperature ( °C )</i>
Up to 70	Below 20
70 to 150	20 to 30

Place a flat cover on top of the receiver to prevent condensed moisture from entering the receiver.

7.3 Apply heat to the flask and adjust the rate so that the first drop of the distillate falls from the end of delivery tube after the interval stated in the specification for the material. When nothing is indicated in the specification for the material, heat the flask at such a rate that the first drop falls into the receiver not less than 5 minutes after heating has begun.

Adjust the heat input so that the distillation proceeds at a rate of 4 to 5 ml per minute ( approximately 2 drops per second ), read the temperature at the instant the first drops falls ( initial boiling point ).

7.4 Record the temperature at the dry point. If a dry point is not obtained ( that is, if active decomposition should occur before the dry-point is reached, as evidenced by a rapid evolution of vapour or heavy fumes, or if there is liquid remaining on the bottom of the flask when the maximum temperature is observed on the thermometer ), record this fact.

7.5 Read and record the barometric pressure.

7.6 If any residue is present, cool to room temperature and pour into a small graduated cylinder. Measure the volume and record it as residue. After the condenser tube has drained, read the total volume and record it as recovery. Record the difference between 100 and the sum of the residue plus recovery as distillation loss.

## **8. CORRECTION TO BE APPLIED TO THE OBSERVED, TEMPERATURES AFTER THE DISTILLATION ( FOR RANGE )**

**8.1 Corrections for Thermometer Error** — If the thermometer gives incorrect readings at the observed initial boiling point or observed dry point, correct the readings by subtracting the amount of error if the thermometer is reading high, or adding the amount of error if the thermometer is reading low.

**8.2 Correction for Barometric Pressure** — When the barometric pressure ( corrected as in 10 ) deviates from 760 mm Hg, apply further corrections to the observed temperatures as indicated in the specification for the -material under test. It should be noted that these last corrections are valid only for pressures above 700 mm Hg.

## **9. FACTORS CAUSING SUPERHEATING**

**9.1** In general, any condition whereby the temperature surrounding the vapour exceeds the temperature of the vapour in equilibrium with the



liquid will cause superheating. Specific factors conducive to superheating are as follows, and should be avoided:

- a) *Flame in contact with the flask* — The applied gas flame should be prevented from contacting more than the specified portion of the flask by the following procedures:
  - 1) Maintain the correct overall dimensions and specified hole diameter of the asbestos board. The hole must be perfectly circular, with no irregularities.
  - 2) Use a board that is free of cracks and checks.
  - 3) Set the flask snugly in the hole in the upper insulating boarding.
- b) *Application of heat* — Attention should be given to burner placement, position, and character of flame, as follows:
  - 1) Apply the source of heat directly beneath the flask. Any variation would result in heating a larger portion of surrounding air to a higher temperature than that of the flask.
  - 2) The flame should not have a larger cross section than is necessary, and should be non-luminous.
  - 3) Place the burner at a level such that the complete combustion area of a non-luminous flame is approximately 20 mm below the board.
- c) *Extraneous heat source* — An extraneous source of heat such as sunlight falling directly on the flask will cause superheating.
- d) *Condition of equipment* — Caution should be observed in employing the apparatus for immediate reuse. For low-boiling materials, cool the heating unit to room temperature before starting the test.
- e) *Use of electric heaters* — Electric heaters will, in general, cause superheating. These should be used only after they have been proven to give results comparable to those obtained when using gas heat. The superheating effect obtained from electric heaters may be minimized, but not completely eliminated, by selecting a heater which, by its design, concentrates the heating elements to a minimum area, and which contains a minimum amount of ceramic material in its overall construction.

## 10. REPORTING

**10.1** If determining distillation yield, report the difference between the volumes of distillate recorded, as the percentage by volume distilling between the specified temperatures at 760 -mm Hg pressure.

**10.2** If determining distillation range, report the corrected initial boiling point and corrected dry point.

## 11. CORRECTIONS TO BAROMETER READING

**11.0** Ascertain the atmospheric pressure by taking a reading on a mercury barometer ( see Note ) having a millimetre scale and applying the following corrections.

**11.1 Index Correction** — Correct the observed barometer reading for index error in accordance with the certificate issued with barometer. The reading thus corrected gives the atmospheric pressure as indicated by a correctly adjusted barometer at the temperature of observation, and under the gravity prevailing at the place of observation, and shall be further corrected to give the equivalent value in millimetres of mercury at 0°C.

**11.2 Temperature Correction to 0°C** — If the mercury barometer is of the **Fortin** type, or of some other type in which the mercury in the cistern is set to a fiducial level when the barometric reading is taken, apply the temperature correction given in Table 1 to the value obtained in 10.1 'index correction'.

**11.2.1** If the barometer used is of the Kew type, that in which the barometric reading is taken without adjusting the level of the mercury in the cistern, the temperature correction will differ somewhat from that given in Table 1. The temperature coefficient of a Kew pattern barometer depends to a small extent on its dimensions, but sufficient -accuracy will be obtained, in general, if the temperature correction to a reading of a Kew pattern barometer is taken to be 5 percent in excess of that given in Table 1 for a **Fortin** barometer.

**NOTE** — Alternatively an aneroid barometer, having a scale calibrated in standard millimetres of mercury, may be employed.

**11.3 Correction to Standard Gravity** — The following information is given for use when distillations are carried out in latitudes where the value of gravity differs widely from the standard value. The reading as corrected in accordance with 11.2 gives the pressure in terms of standard millimetres of mercury at 0°C at the place of observation. To obtain the equivalent pressure at 0°C and under standard gravity multiply the value obtained as above by  $g/980.665$ , where  $g$  is the value of gravity in centimetres per second square at the place of observation.

**TABLE 1 CORRECTION OF BAROMETER READINGS TO 0°C FOR  
FORTIN BAROMETER WITH BRASS SCALE**

( Clause 11.2 )

( The correction is to be subtracted from the barometric reading )

TEMPERATURE OF BAROMETER	BAROMETER READING, mm Hg						
°C	700	720	740	760	780	800	820
10	1.14	1.17	1.21	1.24	1.27	1.30	1.34
11	1.26	1.29	1.33	1.36	1.40		1.47
12		1.41		1.49	1.53	1.44	1.60
13	1.37	1.53	1.45	1.61	1.65	1.70	1.74
14	1.48	1.64	1.57	1.73	1.78	1.88	1.87
15	1.60	1.76	1.69	1.86		1.96	2.00
16	1.71	1.88	1.81	1.98	1.91	2.09	2.14
17	1.82	1.99	1.93	2.10	2.16	2.22	2.27
18	1.94	2.11	2.05	2.23		2.35	2.40
19	2.05	2.23	2.17	2.35	2.29	2.48	2.54
20	2.17	2.34	2.29	2.47	2.41	2.60	2.67
21	2.28	2.46	2.41	2.60	2.54	2.73	2.80
22	2.39	2.58	2.53	2.72	2.67	2.86	2.94
23	2.51	2.69	2.65	2.77	2.79	2.99	3.07
24	2.62	2.81	2.77	2.84	2.92	3.12	3.20
25	2.73		2.89		3.05	3.25	3.33
26	2.85	2.93	3.01	2.97	3.17	3.38	3.47
27	2.96	3.04	3.13	3.09	3.30		3.60
28	3.07	3.16	3.25	3.34	3.42	3.51	3.73
29	3.19	3.28	3.37	3.46	3.55	3.64	
30	3.30	3.39	3.49	3.58	3.63	3.77	3.87
	3.41	3.51	3.61	3.71	3.80	3.90	4.00

Changes in gravity due to causes other than change in latitude, namely, height above sea level, may be neglected and the reduction to standard gravity carried out by means of Table 2 which gives the appropriate corrections for different latitudes. When the sign of the correction in Table 2 is positive add, and when negative subtract, the quantity noted to or from the barometric reading.

**TABLE 2 CORRECTION OF BAROMETRIC READINGS TO STANDARD  
GRAVITY 980.66 cm/s<sup>2</sup>  
( Clause 11.3 )**

LATITUDE DEGREES	BAROMETER READING, mm Hg				
	700	720	740	760	780
0	— 1'88	— 1'93	— 1.99	— 2.04	— 2.10
5	— 1.85	— 1.90	— 1.95	— 2'01	— 2.06
10	— 1.77	— 1.82	— 1.87	— 1.92	— 1.97
15	— 1.63	— 1'68	— 1.72	— 1.77	— 1.82
20	— 1.45	— 1'49	— 1.53	— 1.57	— 1.61
25	— 1.22	— 1.25	— 1.29	— 1.32	— 1.36
30	— 0.96	— 0.98	— 1.01	— 1.04	— 1.07
35	— 0.67	— 0.68	— 0.70	— 0.72	— 0.74
40	— 0.36	— 0.37	— 0.38	— 0.39	— 0.40
45	— 0'04	— 0.04	— 0.04	— 0.04	— 0.04
50	+ 0.29	+ 0.29	+ 0.30	+ 0.31	+ 0.32
55	+ 0.60	+ 0.61	+ 0.62	+ 0.65	+ 0.67
60	+ 0.89	+ 0'91	+ 0.94	+ 0.97	+ 0.99
65	+ 1'15	+ 1'19	+ 1.22	+ 1.25	+ 1.29
70	+ 1.38	+ 1.42	+ 1.46	+ 1.50	+ 1.54

## INTERNATIONAL SYSTEM OF UNITS ( SI UNITS )

Base	Units	
	QUANTITY	UNIT
Length	metre	<b>m</b>
Mass	kilogram	<b>kg</b>
Time	second	<b>s</b>
Electric current	ampere	<b>A</b>
Thermodynamic temperature	kelvin	<b>K</b>
Luminous intensity	candela	<b>cd</b>
Amount of substance	mole	<b>mol</b>

## Supplementary Units

QUANTITY	UNIT	SYSTEM
Plane angle	radian	rad
Solid angle	steradian	sr

## Derived Units

QUANTITY	UNIT	SYMBOL	DEFINITION
Force	newton	<b>N</b>	1 N = 1 kg.m/s <sup>2</sup>
Energy	joule	<b>J</b>	1 J = 1 N.m
Power	watt	<b>W</b>	1 W = 1 J/s
Flux	weber	<b>Wb</b>	1 Wb = 1 V.s
Flux density	tesla	<b>T</b>	1 T = 1 Wb/m <sup>2</sup>
Frequency	hertz	<b>Hz</b>	1 Hz = 1 c/s (s <sup>-1</sup> )
Electric conductance	siemens	<b>S</b>	1 S = 1 A/V
Electromotive force	volt	<b>V</b>	1 V = 1 W/A
Pressure, stress	pascal	<b>Pa</b>	1 Pa = 1 N/m <sup>2</sup>

# INDIAN STANDARDS INSTITUTION

Manak Bhavan, 9 Bahadur Shah Zafar Marg NEW DELHI 110002

**Telephones : 26 60 21, 27 01 31**

Telegram : Manaksanstha

*Regional Offices:*

Telephone

Western : Novelty Chambers, Grant Road	BOMBAY 400007	37 97 29
Eastern : 5 Chowringhee Approach	CALCUTTA 700072	27 50 90
Southern : C.I.T. Campus, Adyar	MADRAS 600113	41 24 42
Northern : B69, Phase VII	S.A.S. NAGAR (MOHALI) 160051	8 78 26

**Branch Offices:**

'Pushpak', Nurmohamed Shaikh Marg, Khanpur	AHMADABAD 380001	2 03 91
'F' Block, Unity Bldg, Narasimharaja Square	BANGALORE 560002	22 48 05
Gangotri Complex, Bhadbhada Road, T.T. Nagar	BHOPAL 462003	6 27 16
22E Kalpana Area	BHUBANESHWAR 751014	5 36 27
5-6-56C L. N. Gupta Marg	HYDERABAD 500001	22 10 83
R 14 Yudhister Marg, C Scheme	JAIPUR 302005	6 98 32
117/418 B Sarvodaya Nagar	KANPUR 208005	4 72 82
Patliputra Induatrial Estate	PATNA 800013	6 28 08
Hantex Bldg ( 2nd Floor ), Rlv Station Road	TRIVANDRUM 605001	32 27

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